The N(5)—N(6) bond length of 1·400 (3) Å has the expected value for a single bond. The hydrazine moiety is slightly rotated with respect to the thiadiazine ring [the torsion angle N(6)—N(5)—C(3)—N(2) is 173°].

The other molecular dimensions are as in (1). The isomer is 4*H* in this case too. van der Waals contacts determine the molecular packing (the intermolecular N···N contact distances are larger than 3·5 Å).

References

ALLÉAUME, M., GULKO, A., HERBSTEIN, F. H., KAPON, M. & MARSH, R. E. (1976). *Acta Cryst.* B**32**, 669–682.

B. A. FRENZ & ASSOCIATES INC. (1980). *SDP-Plus Structure Determination Package*. Version 1·0. Enraf–Nonius, Delft, The Netherlands.

BANDOLI, G. & NICOLINI, M. (1977). *J. Cryst. Mol. Struct* **7**, 229–240.

DESIRAJU, G. R. & KAMALA, R. (1983). *Acta Cryst.* C**39**, 358–360.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

KHAN, M. A., TAYLOR, R. W., LEHN, J. M. & DIETRICH, B. (1988). *Acta Cryst.* C**44**, 1928–1931.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

RAFFA, L., DI BELLA, M., MELEGARI, M. & VAMPA, G. (1962). *Farmaco Ed. Sci.* **17**, 331–339.

RAFFA, L., MELEGARI, M. & VAMPA, G. (1966). *Farmaco Ed. Sci.* **21**, 839–845.

Acta Cryst. (1989). C**45**, 1908–1911

Structures of (±)-*trans*-4-(3-Nitrophenyl)- (I) and (+)-*trans*-4-(4-Fluorophenyl)-2-hydroxy-2-methyl-3,4-dihydro-2*H*,5*H*-pyrano[3,2-*c*][1]benzopyran-5-one (II)

BY VAN HENRY SAVELL JR AND EDWARD J. VALENTE

Department of Chemistry, Mississippi College, Clinton, MS 39058, *USA*

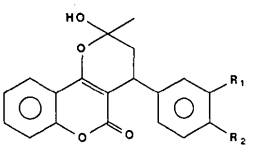
AND DRAKE S. EGGLESTON

Department of Physical and Structural Chemistry, Smith, Kline & French Laboratories, King of Prussia, PA 19406, *USA*

(*Received* 21 *July* 1988; *accepted* 22 *March* 1989)

Abstract. Derivatives of 2-methyl-3,4-dihydro-2*H*,5*H*-pyrano[3,2-*c*][1]benzopyran-5-one. (I) Racemic *trans*-2-hydroxy-4-(3-nitrophenyl), $C_{19}H_{15}NO_6$, $M_r = 353{\cdot}33$, triclinic, $P\bar{1}$, $a = 12{\cdot}468$ (1), $b = 14{\cdot}531$ (3), $c = 9{\cdot}218$ (2) Å, $\alpha = 99{\cdot}00$ (2), $\beta = 98{\cdot}03$ (1), $\gamma = 84{\cdot}35$ (1)°, $V = 1628{\cdot}3$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}441$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 8{\cdot}679$ cm^{-1}, $F(000) = 736$, $T = 294$ K, final $R = 0{\cdot}042$ for 4981 observations [$I \geq 3\sigma(I)$]. The asymmetric unit contains two enantiomeric molecules [(2*R*,4*R*) and (2*S*,4*S*)] unrelated by crystallographic symmetry, and differing slightly in conformation. Hydrogen bonding occurs between a donor hydroxyl on one enantiomer (*A*) and a receptor carbonyl oxygen on another (*B*), O···O = 2·752 (2) Å, and between a donor hydroxyl (*B*) and a receptor hydroxyl (*A*), O···O = 2·923 (2) Å. Dihydropyran rings adopt half-chair conformations distorted toward the *d*,*e*-diplanar conformation. (II) Resolved (2*R*,4*R*)-(+)-*trans*-2-hydroxy-4-(4-fluorophenyl), $C_{19}H_{15}FO_4$, $M_r = 326{\cdot}32$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}838$ (4), $b = 10{\cdot}817$ (3), $c = 14{\cdot}777$ (6) Å, $V = 1572{\cdot}6$ (17) Å^3, $Z = 4$, $D_x = 1{\cdot}374$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 8{\cdot}351$ cm^{-1}, $F(000) = 680$, $T = 294$ K, final $R = 0{\cdot}040$ for all 1583 observations. Hydroxyls are hydrogen bonded intermolecularly to carbonyl groups, O···O = 2·745 (3) Å. The embedded dihydropyran ring adopts a half-chair conformation.

Introduction. Dihydropyran ring conformations can be studied through solid state structures of a series of crystalline warfarin analogs containing the embedded heterocycle. This contribution describes the structures of two substituted 4-aryl derivatives of 2-hydroxy-2-methyl-3,4-dihydro-2*H*,5*H*-pyrano-[3,2-*c*][1]benzopyran-5-one.



(I) $R_1 = NO_2$ $R_2 = H$
(II) $R_1 = H$ $R_2 = F$

Solutions of warfarin analogs consist of an equilibrium mixture of two diastereomeric hemiketals and the intermediate keto form. They crystallize as

0108-2701/89/121908-04$03.00

cyclic hemiketals with axial hydroxyls and the resulting dihydropyran ring may adopt any accessible conformation.

Experimental. Michael-type addition of various unsaturated ketones to 4-hydroxycoumarin leads to the family of warfarin analogs (Bush & Trager, 1983). The 3-nitro derivative was recrystallized from ethyl ethanoate as yellow prisms, m.p. 476–482 K. The 4-fluoro compound was resolved through its quinidine salts (West, Preis, Schroeder & Link, 1961), and the isomer from the more soluble diastereomeric salt was recrystallized from acetone:water producing colorless prisms, m.p. 433–435 K; $[\alpha]_D^{25^\circ C} = +149\,(5)^\circ$, 1·1 g 100 mL^{-1} (0·5 *N* NaOH). Crystallographic specimens: (I) 0·5 × 0·3 × 0·3 mm; (II) 0·6 × 0·2 × 0·25 mm were chosen for data collection on a CAD-4 diffractometer. Unit-cell constants were obtained from a least-squares fit to the angular settings of 25 reflections with $60 < 2\theta < 70^\circ$. Intensities were collected with variable speed θ–2θ scans to $2\theta = 134^\circ$ (*h*: −14 to 14, *k*: −17 to 17, *l*: 0 to 11) for (I) and to 136° (*h*: 0 to 11, *k*: 0 to 13, *l*: 0 to 17) for (II). Three standard intensities monitored every 3 h of exposure time showed a max. change of −0·6 (1·0)% and +1·5 (1·6)%, for (I) and (II) respectively, over the course of data collection; no correction for deterioration was made. The unique data [5539 (I), 1648 (II)] were corrected for coincidence and polarization. To both sets an extinction coefficient of the form proposed by Zachariasen (1963) was applied and refined in the latter stages: $g = 1{\cdot}61\,(1) \times 10^{-6}$ (I), $1{\cdot}54\,(1) \times 10^{-6}$ (II). An empirical absorption correction (Walker & Stuart, 1983) was applied to (I); max./min./av. 1·47/0·68/0·97.

Structures were discovered with *MULTAN* (Germain, Main & Woolfson, 1971). Non-H-atom positions were refined with their U_{iso}'s by full-matrix least squares on *F* minimizing $\sum w(|F_o| - |F_c|)^2$, then with their U_{ij}'s. Except for the hydroxyl H's, H-atom positions were calculated and placed 1·0 Å from their attached atom and were assigned *B*'s approximately 1·3 times the B_{eq} of the adjacent carbon; they were not refined. Scattering factors were from *International Tables for X-ray Crystallography* (1974) except for H (Stewart, Davidson & Simpson, 1965). Final agreement factors for (I): $R = 0{\cdot}042$, $wR = 0{\cdot}069$, GOF = 1·967, and for (II): $R = 0{\cdot}037$, $wR = 0{\cdot}046$, GOF = 2·398 for the 4981 and 1451 intensities greater than three times their estimated standard deviations, for (I) and (II) respectively. A weighting scheme of the type $w = 4F_o^2/s^2(I)$ with $s(I) = [\sigma(I)^2 + 0{\cdot}03(F_o)^2]^{1/2}$ and $\sigma(I)$ from counting statistics was used in each refinement. There were 590 (I) and 278 (II) variables; scale factors 0·071 (I) and 0·090 (II); final maximum Δ/σ less than 0·07 for each structure; maximum positive and negative excursions less than 0·18 and 0·12 e Å^{-3}. Final atom positions and equivalent isotropic vibrational factors for the non-H atoms are given in Tables 1–3. Principal bond distances for both (I) and (II) are found in Table 4.* All programs used were from the locally modified Enraf–Nonius (1979) *SDP*.

Table 1. *Positions and B_{eq}'s for* (I), *molecule A, with e.s.d.'s in parentheses*

	x	*y*	*z*	B_{eq}(Å^2)*
N1*A*	−0·6832 (2)	0·8752 (1)	0·1620 (3)	6·95 (5)
O1*A*	−0·3259 (1)	0·55516 (8)	0·0754 (1)	4·02 (2)
O2*A*	−0·2797 (1)	0·68587 (9)	0·2154 (1)	4·59 (3)
O3*A*	−0·35820 (9)	0·67743 (7)	−0·3026 (1)	3·37 (2)
O4*A*	−0·19499 (9)	0·74666 (8)	−0·2590 (1)	3·78 (2)
O5*A*	−0·7381 (2)	0·9202 (2)	0·2495 (3)	13·01 (1)
O6*A*	−0·7188 (1)	0·8171 (2)	0·0642 (3)	9·71 (6)
C2*A*	−0·3114 (1)	0·6493 (1)	0·0920 (2)	3·38 (3)
C3*A*	−0·3329 (1)	0·6959 (1)	−0·0380 (2)	2·89 (3)
C4*A*	−0·3547 (1)	0·6442 (1)	−0·1731 (2)	2·87 (3)
C5*A*	−0·4201 (1)	0·4969 (1)	−0·3203 (2)	3·90 (3)
C6*A*	−0·4492 (2)	0·4077 (1)	−0·3236 (2)	4·54 (4)
C7*A*	−0·4347 (2)	0·3675 (1)	−0·1946 (2)	4·78 (4)
C8*A*	−0·3917 (2)	0·4162 (1)	−0·0616 (2)	4·50 (4)
C9*A*	−0·3658 (1)	0·5069 (1)	−0·0590 (2)	3·44 (3)
C10*A*	−0·3785 (1)	0·5484 (1)	−0·1865 (2)	3·16 (3)
C11*A*	−0·3217 (1)	0·7995 (1)	−0·0192 (2)	3·05 (3)
C12*A*	−0·3495 (1)	0·8335 (1)	−0·1703 (2)	3·47 (3)
C13*A*	−0·3074 (1)	0·7644 (1)	−0·2945 (2)	3·19 (3)
C14*A*	−0·3388 (2)	0·7941 (1)	−0·4450 (2)	4·00 (4)
C15*A*	−0·3954 (1)	0·8554 (1)	0·0869 (2)	3·08 (3)
C16*A*	−0·5044 (1)	0·8401 (1)	0·0745 (2)	3·72 (3)
C17*A*	−0·5678 (1)	0·8924 (1)	0·1743 (2)	4·21 (4)
C18*A*	−0·5280 (2)	0·9598 (1)	0·2846 (2)	4·66 (4)
C19*A*	−0·4216 (2)	0·9774 (1)	0·2919 (2)	5·15 (4)
C20*A*	−0·3555 (2)	0·9258 (1)	0·1942 (2)	4·29 (4)

* Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

Discussion. The crystal structure of racemic 3-nitrowarfarin (I) contains the enantiomers [(2*R*,4*R*) and (2*S*,4*S*)] in the asymmetric unit, unrelated by crystallographic symmetry. Intermolecular associations differ for the two; molecule *A* donates a hydrogen bond through hydroxyl O4 to the carbonyl oxygen O2 of molecule *B*, the O4*A*···O2*B* separation is 2·750 (2) Å, the H4*A*···O2*B* separation is 1·82 (2) Å and the angle at hydrogen is 168 (2)°. Molecule *B* donates a hydrogen bond through O4 but the acceptor is the hydroxyl O4 of molecule *A*. The O4*B*···O4*A* separation is 2·921 (2) Å, the H4*B*···O4*A* separation is 2·07 (2) Å and the angle at hydrogen is 177 (2)°. Thus O4*A* forms two hydrogen bonds, O4*B* and O2*B* each one, and O2*A* none. The torsion angle O3/C13/O4/H4 is 91 (2)° in *A* and 62 (2)° in *B*, the largest conformational difference between the two enantiomers, which are otherwise quite similar. Each of the 4-(3-nitrophenyl) groups are disposed pseudo-

* Lists of principal bond angles, H-atom positions, anisotropic vibrational amplitudes and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52069 (46 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Positions and B_{eq}'s for* (I), *molecule B, with e.s.d.'s in parentheses*

	x	y	z	B_{eq}(Å²)*
N1*B*	−0·3437 (1)	0·2895 (1)	0·3575 (2)	4·22 (3)
O1*B*	0·09698 (9)	0·15333 (8)	0·5357 (1)	3·86 (2)
O2*B*	0·0725 (1)	0·29812 (9)	0·4900 (1)	4·11 (2)
O3*B*	−0·01153 (9)	0·22731 (7)	0·9415 (1)	3·39 (2)
O4*B*	0·07585 (9)	0·36161 (8)	1·0376 (1)	3·64 (2)
O5*B*	−0·3524 (1)	0·2213 (1)	0·4163 (2)	6·09 (4)
O6*B*	−0·3978 (1)	0·3045 (1)	0·2414 (2)	6·48 (4)
C2*B*	0·0612 (1)	0·2448 (1)	0·5750 (2)	3·20 (3)
C3*B*	0·0139 (1)	0·2713 (1)	0·7108 (2)	2·85 (3)
C4*B*	0·0189 (1)	0·2081 (1)	0·8058 (2)	2·88 (3)
C5*B*	0·0604 (2)	0·0420 (1)	0·8535 (2)	4·82 (4)
C6*B*	0·0964 (2)	−0·0487 (1)	0·8033 (3)	5·94 (5)
C7*B*	0·1295 (2)	−0·0711 (1)	0·6640 (3)	5·51 (5)
C8*B*	0·1295 (1)	−0·0040 (1)	0·5749 (2)	4·61 (4)
C9*B*	0·0933 (1)	0·0874 (1)	0·6259 (2)	3·52 (3)
C10*B*	0·0581 (1)	0·1117 (1)	0·7636 (2)	3·38 (3)
C11*B*	−0·0356 (1)	0·3701 (1)	0·7442 (2)	2·97 (3)
C12*B*	−0·0915 (1)	0·3789 (1)	0·8853 (2)	3·38 (3)
C13*B*	−0·0262 (1)	0·3270 (1)	1·0024 (2)	3·15 (3)
C14*B*	−0·0837 (2)	0·3276 (1)	1·1364 (2)	4·31 (4)
C15*B*	−0·1182 (1)	0·3963 (1)	0·6172 (2)	2·96 (3)
C16*B*	−0·1935 (1)	0·3334 (1)	0·5479 (2)	3·19 (3)
C17*B*	−0·2668 (1)	0·3572 (1)	0·4310 (2)	3·39 (3)
C18*B*	−0·2716 (1)	0·4426 (1)	0·3806 (2)	3·79 (3)
C19*B*	−0·1987 (1)	0·5059 (1)	0·4524 (2)	3·82 (3)
C20*B*	−0·1231 (1)	0·4826 (1)	0·5683 (2)	3·57 (3)

* Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

Table 3. *Positions and B_{eq}'s for* (II) *with e.s.d.'s in parentheses*

	x	y	z	B_{eq}(Å²)*
F1	−0·2276 (2)	0·8406 (2)	0·6488 (1)	9·19 (5)
O1	0·3230 (2)	1·0900 (2)	0·9354 (1)	5·44 (4)
O2	0·1256 (2)	0·9985 (2)	0·9435 (1)	5·81 (4)
O3	0·5192 (2)	0·7648 (1)	0·8755 (1)	4·31 (3)
O4	0·4174 (2)	0·6253 (2)	0·9724 (1)	5·28 (4)
C2	0·2453 (3)	0·9859 (2)	0·9255 (2)	4·72 (5)
C3	0·3098 (3)	0·8734 (2)	0·8965 (2)	4·03 (5)
C4	0·4478 (3)	0·8685 (2)	0·8930 (2)	3·96 (5)
C5	0·6709 (3)	0·9806 (3)	0·9006 (2)	5·14 (6)
C6	0·7406 (3)	1·0908 (3)	0·9119 (2)	5·86 (7)
C7	0·6699 (3)	1·1982 (2)	0·9301 (2)	5·53 (7)
C8	0·5299 (3)	1.1984 (2)	0·9372 (2)	5·67 (7)
C9	0·4624 (3)	1·0873 (2)	0·9252 (2)	4·63 (6)
C10	0·5294 (3)	0·9780 (2)	0·9067 (2)	4·15 (5)
C11	0·2218 (3)	0·7615 (2)	0·8775 (2)	4·30 (5)
C12	0·3105 (3)	0·6620 (2)	0·8337 (2)	4·64 (6)
C13	0·4438 (3)	0·6473 (2)	0·8823 (2)	4·34 (5)
C14	0·5379 (3)	0·5535 (2)	0·8397 (2)	5·32 (6)
C15	0·1006 (3)	0·7867 (2)	0·8168 (2)	4·33 (5)
C16	0·1101 (3)	0·8622 (3)	0·7413 (2)	5·72 (7)
C17	−0·0000 (4)	0·8804 (3)	0·6840 (2)	6·53 (8)
C18	−0·1176 (3)	0·8202 (3)	0·7037 (2)	6·11 (7)
C19	−0·1327 (3)	0·7451 (3)	0·7751 (2)	6·52 (7)
C20	−0·0208 (3)	0·7286 (3)	0·8332 (2)	5·40 (6)

* Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

equatorially, and the dihedral angles between coumarin and phenyl planes are 102 and 109°. Conformations of the dihydropyran rings approach the *d,e*-diplanar form. Ring displacement asymmetry parameters ΔC_2 are (*A*) 0·0375 (7) and (*B*) 0·0183 (7) (Nardelli, 1983). Dihydropyran torsion angles are given in Table 5; *ORTEP* drawings (Johnson, 1976) are given in Figs. 1 and 2.

The crystal structure of (*R*)-(+)-4′-fluorowarfarin (II) is pseudoisomorphous with that of (*S*)-(−)-warfarin (Valente, Trager & Jensen, 1975). Hydrogen

Table 4. *Principal bond distances* (Å) *for* (I) *and* (II)

(I)

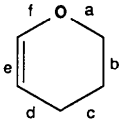
N1*A*—O5*A*	1·203 (2)	N1B—O5*B*	1·223 (2)
N1*A*—O6*A*	1·203 (2)	N1B—O6*B*	1·221 (2)
N1*A*—C17*A*	1·470 (2)	N1B—C17*B*	1·463 (2)
O1*A*—C2*A*	1·380 (2)	O1*B*—C2*B*	1·369 (1)
O1*A*—C9*A*	1·377 (1)	O1B—C9*B*	1·372 (2)
O2*A*—C2*A*	1·208 (1)	O2*B*—C2*B*	1·216 (2)
O3*A*—C4*A*	1·349 (1)	O3*B*—C4*B*	1·338 (1)
O3*A*—C13*A*	1·456 (1)	O3*B*—C13*B*	1·472 (1)
O4*A*—C13*A*	1·402 (1)	O4*B*—C13*B*	1·392 (1)
C2*A*—C3*A*	1·447 (2)	C2*B*—C3*B*	1·440 (1)
C3*A*—C4*A*	1·357 (1)	C3*B*—C4*B*	1·357 (2)
C3*A*—C11*A*	1·506 (2)	C3*B*—C11*B*	1·509 (2)
C4*A*—C10*A*	1·436 (2)	C4*B*—C10*B*	1·449 (2)
C5*A*—C6*A*	1·375 (2)	C5*B*—C6*B*	1·380 (2)
C5*A*—C10*A*	1·399 (2)	C5*B*—C10*B*	1·401 (2)
C6*A*—C7*A*	1·387 (2)	C6*B*—C7*B*	1·384 (3)
C7*A*—C8*A*	1·382 (2)	C7*B*—C8*B*	1·370 (3)
C8*A*—C9*A*	1·383 (2)	C8*B*—C9*B*	1·390 (2)
C9*A*—C10*A*	1·386 (2)	C9*B*—C10*B*	1·383 (2)
C11*A*—C12*A*	1·533 (2)	C11*B*—C12*B*	1·541 (2)
C11*A*—C15*A*	1·521 (1)	C11*B*—C15*B*	1·516 (2)
C12*A*—C13*A*	1·516 (2)	C12*B*—C13*B*	1·511 (2)
C13*A*—C14*A*	1·506 (2)	C13*B*—C14*B*	1·511 (2)
C15*A*—C16*A*	1·385 (2)	C15*B*—C16*B*	1·394 (2)
C15*A*—C20*A*	1·386 (2)	C15*B*—C20*B*	1·391 (2)
C16*A*—C17*A*	1·384 (2)	C16*B*—C17*B*	1·377 (2)
C17*A*—C18*A*	1·372 (2)	C17*B*—C18*B*	1·384 (2)
C18*A*—C19*A*	1·366 (2)	C18*B*—C19*B*	1·385 (2)
C19*A*—C20*A*	1·387 (2)	C19*B*—C20*B*	1·383 (2)

(II)

F1—C18	1·371 (3)	C7—C8	1·381 (3)
O1—C2	1·369 (2)	C8—C9	1·384 (3)
O1—C9	1·381 (2)	C9—C10	1·381 (3)
O2—C2	1·215 (2)	C11—C12	1·530 (3)
O3—C4	1·348 (2)	C11—C15	1·517 (3)
O3—C13	1·475 (2)	C12—C13	1·504 (3)
O4—C13	1·378 (2)	C13—C14	1·511 (2)
C2—C3	1·438 (3)	C15—C16	1·386 (2)
C3—C4	1·359 (2)	C15—C20	1·371 (3)
C3—C11	1·515 (2)	C16—C17	1·389 (3)
C4—C10	1·445 (2)	C17—C18	1·359 (3)
C5—C6	1·385 (3)	C18—C19	1·339 (3)
C5—C10	1·395 (3)	C19—C20	1·408 (3)
C6—C7	1·381 (3)		

Table 5. *Torsion angles* (°) *in the dihydropyran rings*

E.s.d.'s are at most about 0·4°; common configuration.



	a	*b*	*c*	*d*	*e*	*f*
(I)*A*	−49·2	60·9	−38·6	5·4	6·5	17·0
(I)*B*	−46·3	54·6	−40·2	8·4	5·1	15·0
(II)	−46·1	62·5	−45·4	13·6	2·3	14·4

bonding occurs between screw related molecules along the *a* axis with donor O4 hydroxyls and receptor carbonyl O2's, O4···O2 = 2·745 (3), H4···O2 = 1·78 Å with an O4—H4···O2 angle of 170 (3)°. In the molecular structure, the 4-fluorophenyl group is disposed pseudoequatorially and is inclined at 69° (dihedral angle) with respect to the coumarin plane. The embedded dihydropyran ring adopts an almost undistorted half-chair conformation, ΔC_2 = 0·006 (2); intra-ring torsion angles are given in Table 5. A drawing of the structure is presented in Fig. 3.

This work was sponsored in part by a grant from the American Heart Association, No. MS-86-G-4.

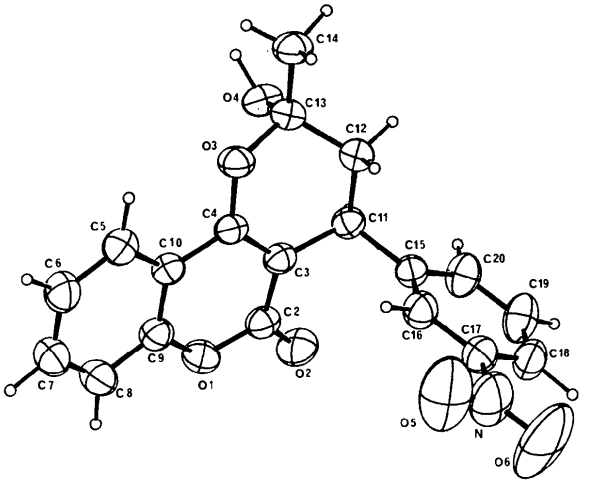


Fig. 1. A drawing of (I) showing the *A* molecule with 50% probability ellipsoids for the non-H atoms.

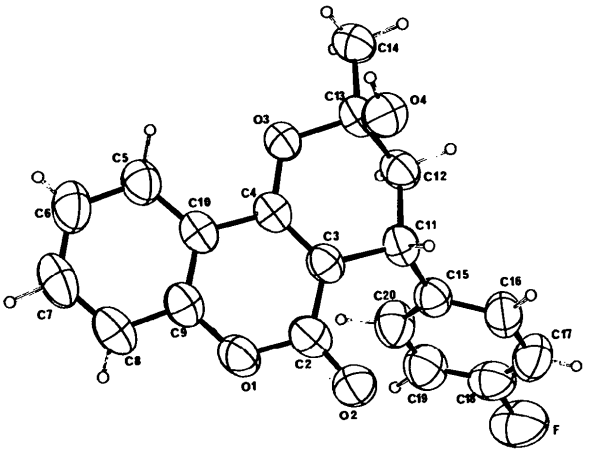


Fig. 3. A drawing of (II) with 50% probability ellipsoids for the non-H atoms.

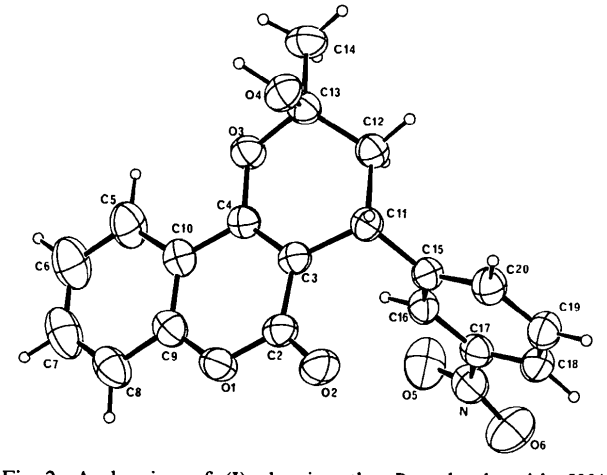


Fig 2. A drawing of (I) showing the *B* molecule with 50% probability ellipsoids for the non-H atoms.

References

BUSH, E. & TRAGER, W. F. (1983). *J. Pharm. Sci.* **72**, 830–831.

Enraf–Nonius (1979). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

NARDELLI, M. (1983). *Acta Cryst.* C**39**, 1141–1142.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

VALENTE, E. J., TRAGER, W. F. & JENSEN, L. H. (1975). *Acta Cryst.* B**31**, 954–960.

WALKER, N. & STUART, D. (1983). *Acta Cryst.* A**39**, 158–166.

WEST, B. D., PREIS, S., SCHROEDER, C. & LINK, K. P. (1961). *J. Am. Chem. Soc.* **83**, 2676–2679.

ZACHARIASEN, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1989). C**45**, 1911–1914

Structure of [1,1′:4′(*trans*),1″:4″(*trans*),1‴-Quatercyclohexane]-1″,4′-diamine

BY CLAUS KRIEGER, ELFRIEDE M. WILL AND FRANZ A. NEUGEBAUER*

Max-Planck-Institut für medizinische Forschung, Abteilung Organische Chemie, Jahnstr. 29, *D*-6900 *Heidelberg, Federal Republic of Germany*

(*Received* 24 *January* 1989; *accepted* 29 *March* 1989)

Abstract. $C_{24}H_{44}N_2$, $M_r = 360{\cdot}63$, triclinic, $P\bar{1}$, $a = 6{\cdot}024$ (1), $b = 10{\cdot}763$ (2), $c = 17{\cdot}159$ (3) Å, $\alpha = 78{\cdot}54$ (2), $\beta = 81{\cdot}97$ (2), $\gamma = 90{\cdot}00$ (2)°, $V = 1079{\cdot}1$ (6) Å^3, $Z = 2$, $D_x = 1{\cdot}111$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·594 cm^{-1}, $F(000) = 404$, $T = 295$ K, $R = 0{\cdot}049$ for 2674 unique observed reflections [$I \geq 2\sigma(I)$]. The crystal structure shows an equatorial alignment of all four cyclohexane rings and confirms the axial position of both amino groups. In the crystal there are two independent molecules: *A* with a 'parallel' alignment of all cyclohexane rings, showing a twist angle between central

* To whom correspondence should be addressed.

0108-2701/89/121911-04$03.00